

LA-UR-18-24880

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Title: RBS uncertainty study

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Intended for: Report

Issued: 2018-06-06

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RBS uncertainty study

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05-22-18

LA-UR-18-xxxx



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“Review of RBS measurements accuracy and recommendations to improve it”, I. Usov et al., LA-UR-17-31226

RBS strengths and weaknesses from a customer viewpoint

Strengths

Quantitative and non-destructive analysis of thin films and near surface regions of bulk materials:

- Chemical composition and Impurity elements
- Stoichiometry and Areal elemental density
- Depth profiling and Thickness

Weaknesses

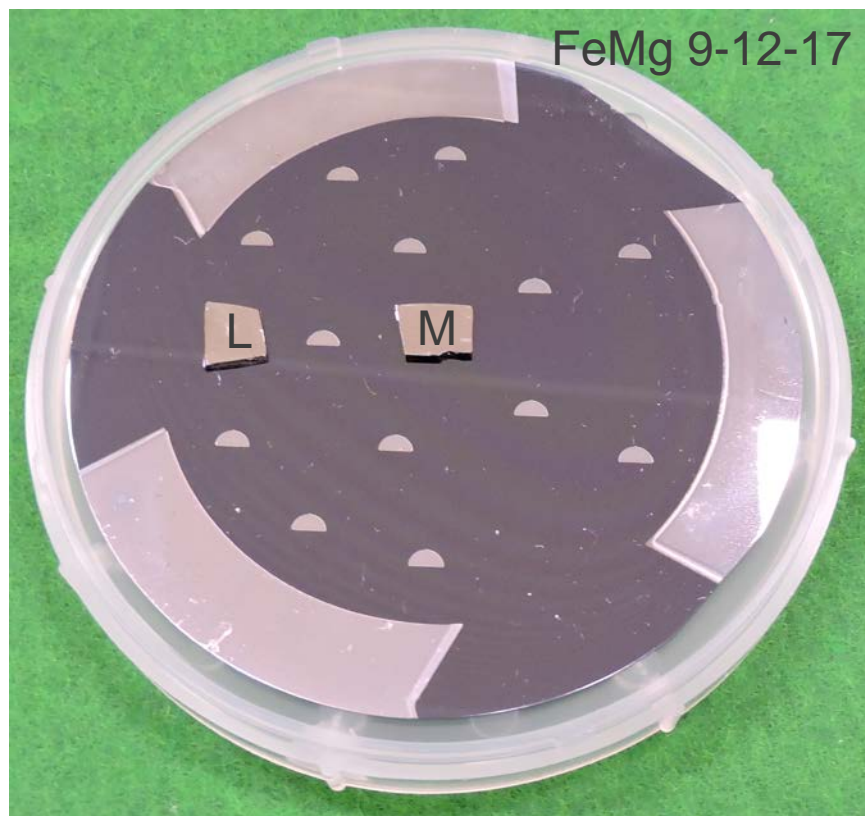
- High cost (1-3K per sample)
- Expertize in RBS is not widely available
- Experimental set up is often one of a kind “home made thing”
- Discrepancies between data are possible

RBS measurements can be VERY accurate

<i>Sources of RBS data uncertainties</i>	<i>Cures</i>
1 st : instrument/operator	Maintenance, calibration, upgrades, expertise
2 nd : stopping cross section	Do not rely on TRIM. Measure it.
3 rd : sample quality	Choose alloys that can actually be fabricated
4 th : data analysis	Software, expertise and diligence

Independent RBS measurements are often used to standardize procedures
RBS accuracy: ~ 1% is possible

Typical set of opacity foils and witness samples



Opacity foil section view

top parylene ($\sim 10\ \mu\text{m}$)

target alloy ($\sim 0.3\ \mu\text{m}$)

bottom parylene ($\sim 10\ \mu\text{m}$)

Si wafer

Witness section view

target alloy ($\sim 0.3\ \mu\text{m}$)

Carbon
substrate

13 opacity foils and 2 witnesses (L-left and M-middle) were selected for independent RBS analysis at LANL, LLNL and EAG

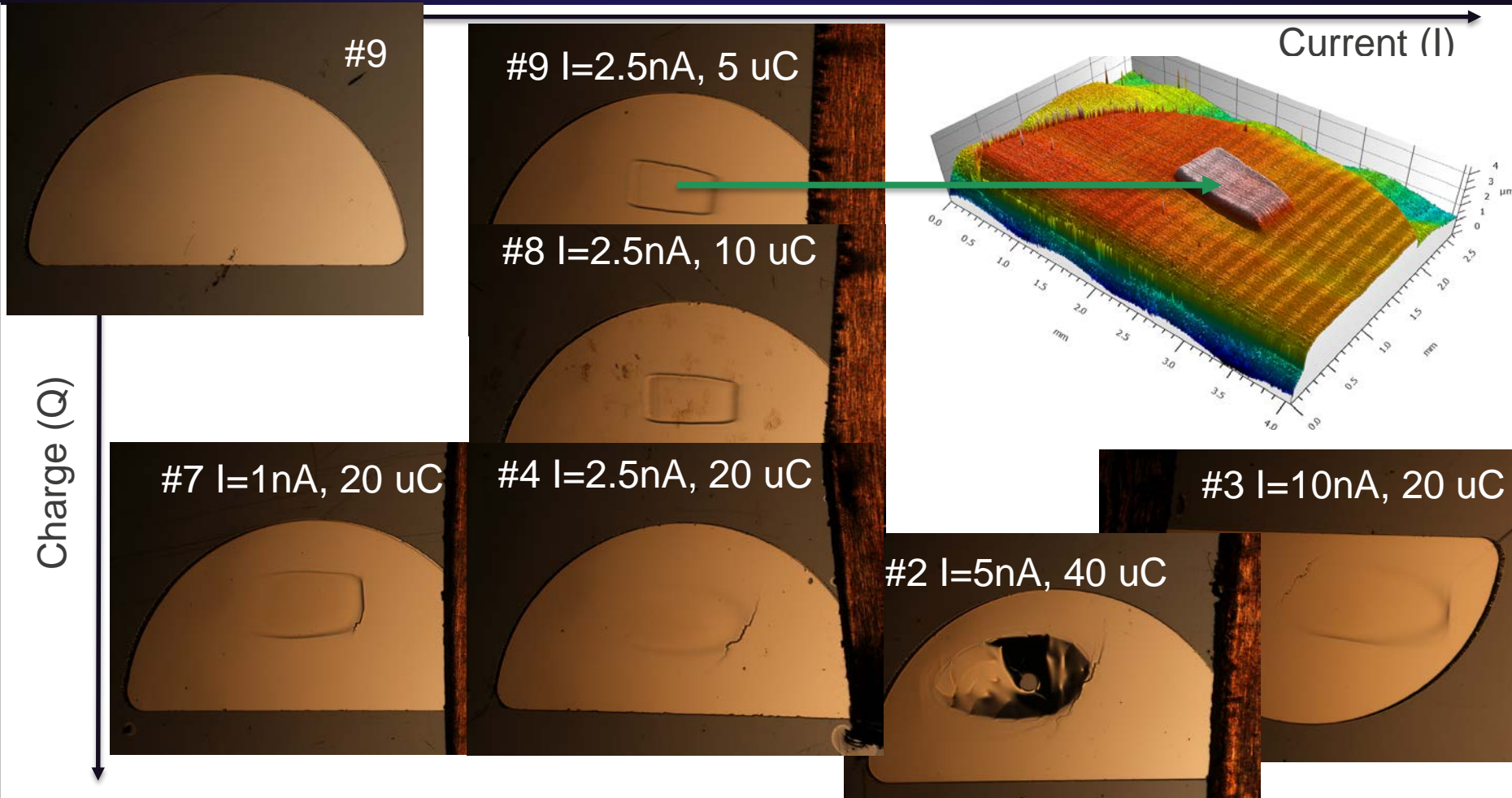


RBS measurements conditions and data analysis

	LANL	LLNL	EAG
samples	#1- #9 and 2 witness	10-13	10,11,13
Ion	He++	He+	He++
Energy	3	3	2.275
Current	1 - 10 nA	1 - 5 nA	10 nA
Charge	5 – 40 μC	0.1 – 5 μC	40
Geometry	Cornell	IBM	IBM
Backscattering angle	167	165.43	160 and 112
Data analysis software	RUMP, SIMNRA	RUMP, SIMNRA	Proprietary code (looks like SIMNRA)

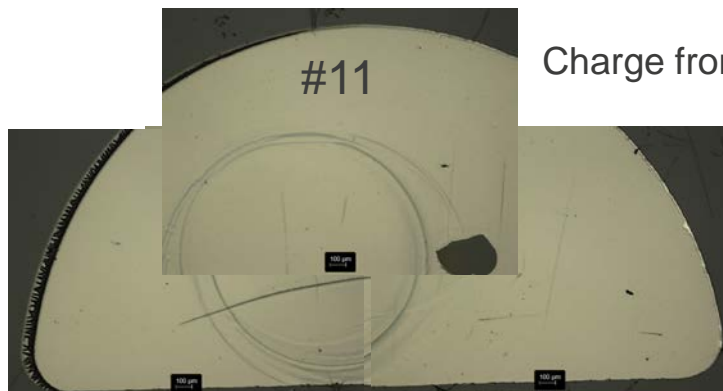
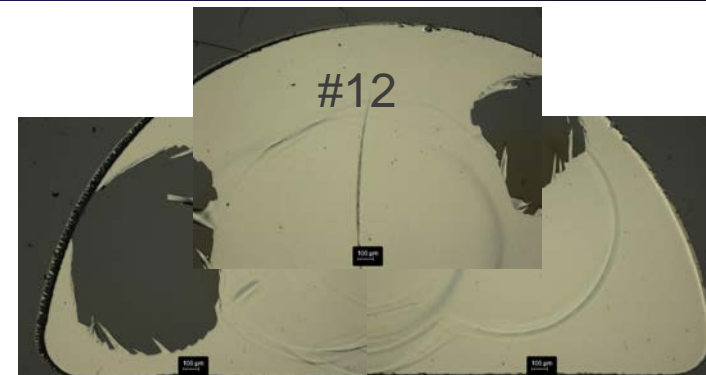
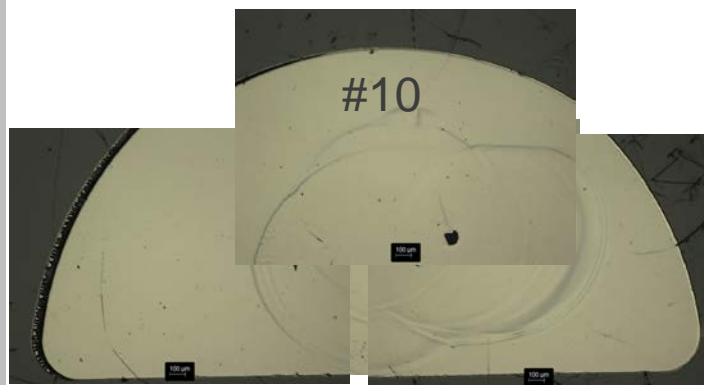


RBS conditions (beam current and charge) optimization at IBML (LANL)

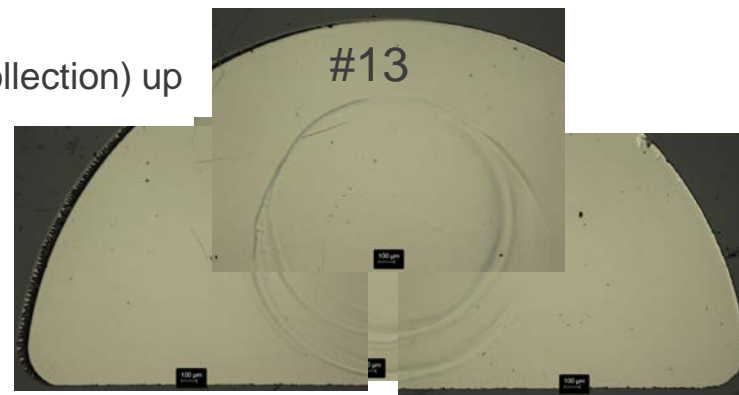


Nikon 20X images and Nanovea 3D height map: area analyzed by RBS (exposed to 3MeV He ions) is raised by $\sim 1 \mu\text{m}$ above the foil surface

RBS conditions (beam current and charge) optimization at LLNL



Current ~1-5 nA,
Charge from ~0.1 μC (<100s collection) up
to ~5 μC (~1000s)



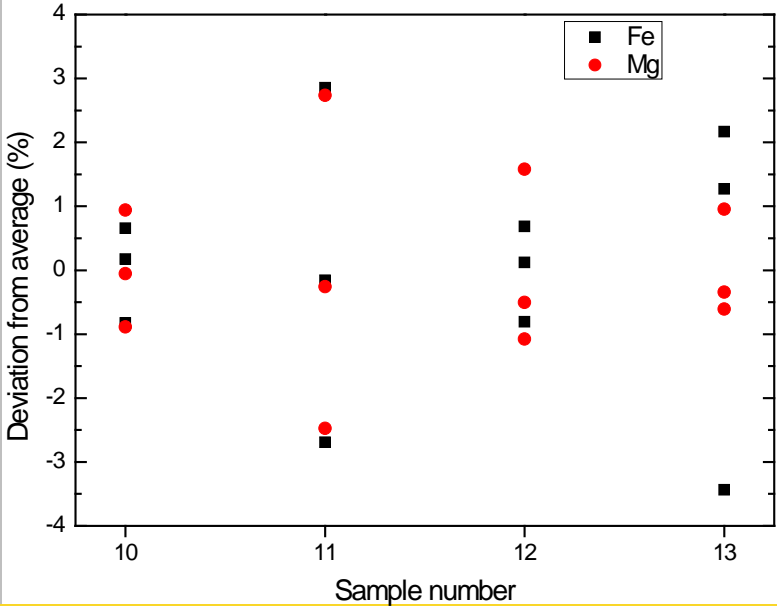
Observations by Swanee:

- All films survived except for near the film edge of #12.
- #12 was ok at ~0.2 μC (for 5 times), but I observed one spot near the film edge started to delaminate when I irradiated ~2 μC at once, and another spot near the film edge at the next ~2 μC irradiation on slightly different (fresh) area.

Delamination occurred between 1 and 3 μC

LLNL: Areal density uncertainty from 3 independent measurements taken from the same sample

	#10		#11		#12		#13	
	[Fe]	[Mg]	[Fe]	[Mg]	[Fe]	[Mg]	[Fe]	[Mg]
1	3.330	1.930	3.410	1.854	3.376	1.909	3.427	1.873
2	3.281	1.895	3.310	1.760	3.357	1.898	3.397	1.844
3	3.314	1.911	3.226	1.800	3.326	1.949	3.239	1.849
Average	3.308	1.912	3.315	1.805	3.353	1.919	3.354	1.855



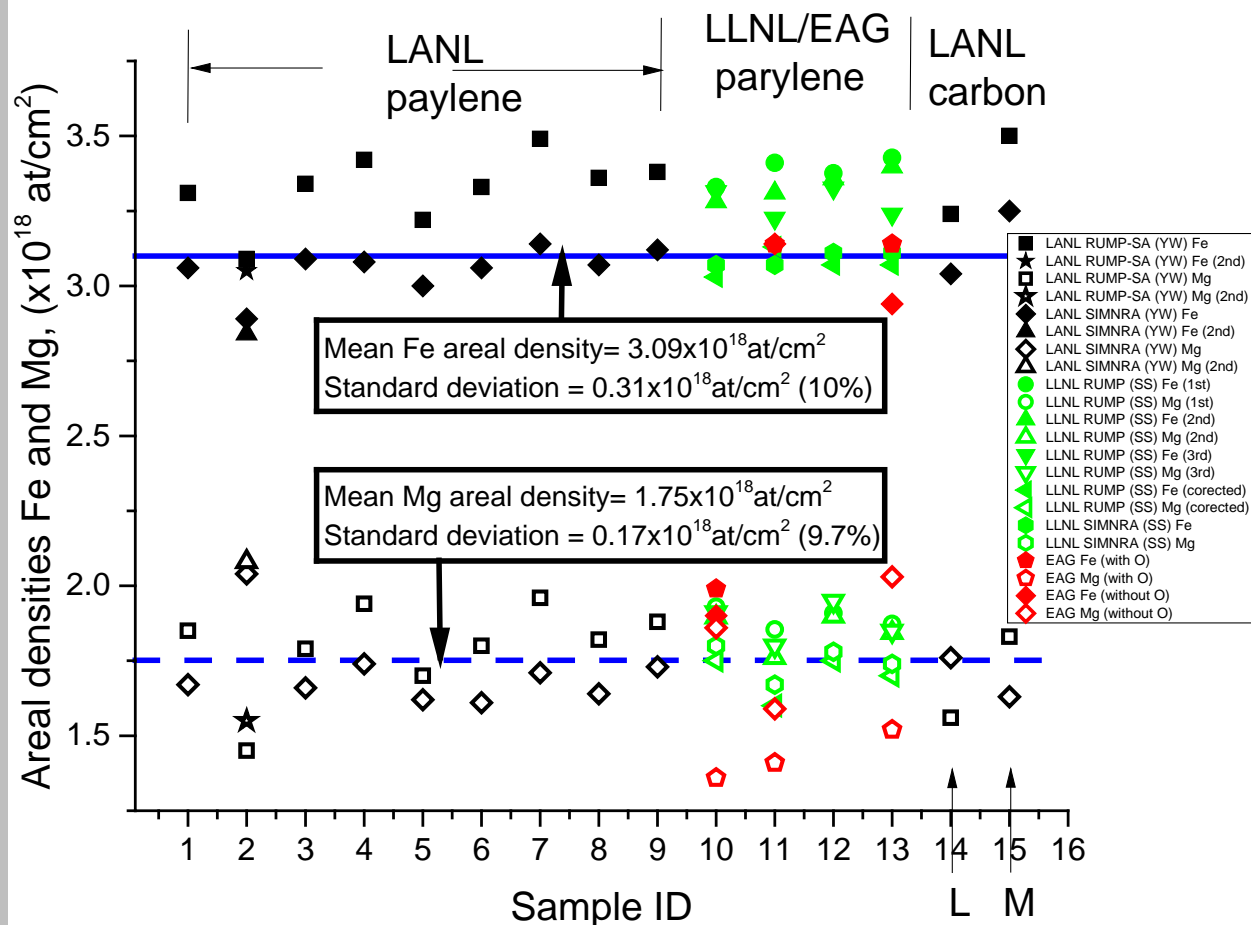
geom ibm
energy 3.0
theta 0
phi 14.57
beam 4He+
Conversion 2.893 51.96
omega 14
FWHM 15.000000
Current 0.5
Tau 5.0

corr 1.395
ch 0.5

At 0.5 μC , counting statistics for Mg is 18% and for Fe is ~ 6%



Comparison of ALL Fe and Mg areal densities measured by RBS at LANL, LLNL and EAG



Sources of RBS data uncertainties

1st: instrument/operator

2nd: stopping cross section

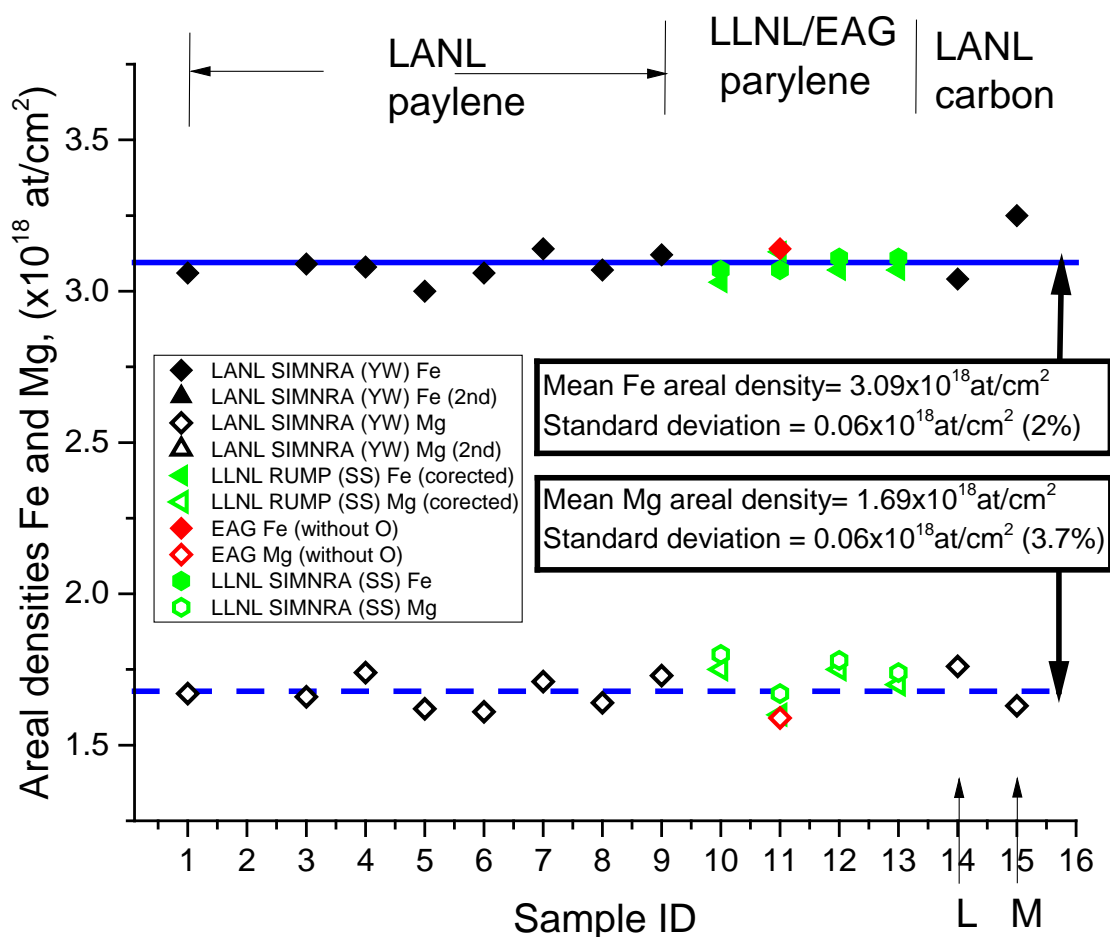
3rd: sample quality

4th: data analysis

Standard deviation of 10% was found for RBS data with 2nd, 3rd and 4th uncertainties



Comparison of SELECTED Fe and Mg areal densities measured by RBS at LANL, LLNL and EAG



Opacity alloy on carbon witness vs paylene

Deviation from paylene average:

FeMg-L: Fe(1.6%); Mg(4.1%)
FeMg-M: Fe(5.2%); Mg(3.6%)

Standard deviations for Fe and Mg areal densities (on paylene) were found to be 2.0% and 3.7%, respectively.



Fe and Mg areal densities measured by RBS at LANL, LLNL and EAG

ID	Elements	Nominal composition, at/cm ²	RUMP-SA (YW), at/cm ²	SIMNRA (YW), at/cm ²	LLNL RUMP (SS), at/cm ²	LLNL RUMP corrected (SS), at/cm ²	LLNL SIMNRA (SS), at/cm ²	EAG (C0JZA638 22 March 2018)	EAG refit without Oxygen (C0JZA638 May 2, 2018)
FeMg-L	Fe	3.00E+18	3.24E+18	3.04E+18					
	Mg	1.50E+18	1.56E+18	1.76E+18					
FeMg_M	Fe	3.00E+18	3.50E+18	3.25E+18					
	Mg	1.50E+18	1.83E+18	1.63E+18					
FeMg_1	Fe	3.00E+18	3.31E+18	3.06E+18					
	Mg	1.50E+18	1.85E+18	1.67E+18					
FeMg_2 (2nd run)	Fe	3.00E+18	3.05E+18	2.84E+18					
	Mg	1.50E+18	1.55E+18	2.08E+18					
FeMg_2 (1st run)	Fe	3.00E+18	3.09E+18	2.89E+18					
	Mg	1.50E+18	1.45E+18	2.04E+18					
FeMg_3	Fe	3.00E+18	3.34E+18	3.09E+18					
	Mg	1.50E+18	1.79E+18	1.66E+18					
FeMg_4	Fe	3.00E+18	3.42E+18	3.08E+18					
	Mg	1.50E+18	1.94E+18	1.74E+18					
FeMg_5	Fe	3.00E+18	3.22E+18	3.00E+18					
	Mg	1.50E+18	1.70E+18	1.62E+18					
FeMg_6	Fe	3.00E+18	3.33E+18	3.06E+18					
	Mg	1.50E+18	1.80E+18	1.61E+18					
FeMg_7	Fe	3.00E+18	3.49E+18	3.14E+18					
	Mg	1.50E+18	1.96E+18	1.71E+18					
FeMg_8	Fe	3.00E+18	3.36E+18	3.07E+18					
	Mg	1.50E+18	1.82E+18	1.64E+18					
FeMg_9	Fe	3.00E+18	3.38E+18	3.12E+00					
	Mg	1.50E+18	1.88E+18	1.73E+18					
FeMg_10	Fe	3.00E+18			3.31E+18	3.03e18	3.07e18	1.99E+18	1.90e18
	Mg	1.50E+18			1.91E+18	1.75e18	1.80e18	1.36E+18	1.86e18
	O							2.63E+17	No O, C=34.5% and Si=6.8%
FeMg_11	Fe	3.00E+18			3.32E+18	3.3e18	3.07e18	3.14E+18	3.14e18
	Mg	1.50E+18			1.81E+18	1.65e18	1.67e18	1.41E+18	1.59e18
	O							1.90E+17	0
FeMg_12	Fe	3.00E+18			3.35E+18	3.07e18	3.11e18		
	Mg	1.50E+18			1.92E+18	1.75e18	1.78e18		
	O								
FeMg_13	Fe	3.00E+18			3.35E+18	3.07e18	3.11e18	3.14E+18	2.94e18
	Mg	1.50E+18			1.86E+18	1.70e18	1.74e18	1.52E+18	2.03e18
	O							2.45E+17	0



Thoughts, Conclusions and Future work

Bob Heeter's thoughts:

- ❑ 7% areal density uncertainty is the nominal requirement from the error budgeting. A tighter uncertainty would be beneficial for the long term.
- ❑ The sample metrology must include all materials present in the sample, e.g. including oxygen as well as iron-magnesium or barium-aluminum, since the X-ray transmission through the (hot or cold) sample is affected by whatever is in the sample, but not the tamper. So we are very interested in finding a way to accurately infer impurity content.

Conclusions on Fe and Mg areal density uncertainties:

- ❑ 3% (or less) for 3 independent measurements taken from the same sample at LLNL
- ❑ 2% (Fe) and 3.7% (Mg) for measurements performed at LANL, LLNL and EAG
- ❑ 5.2% (or less for Fe) and 4.1% (or less for Mg) deviation between the witness and opacity foil

Future work:

- ❑ Determine RBS measurements absolute accuracy for Fe and Mg
- ❑ Compare RBS areal densities with values found with X-ray absorption techniques:
 - Edge (GA)
 - DSC (LANL)
- ❑ Impurity (mainly Oxygen) content measurements
- ❑ Determine FeMg deposition uniformity over 4" wafer